

# Materials Compatibility

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# Overview

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## Timeline

- Project start date Oct 2003
- Project end date Sep 2015
- Percent complete 33%

## Budget

- Total project funding (from FY03)
  - DOE share: \$8.3M
- FY06 Funding: \$1.5M
- FY07 Funding: \$2.9M (\$800K for materials compatibility)

## Partners

- Collaborators:
  - ASME
  - CSA
  - Swagelok
  - NIST
  - DOE Pipeline Working Group

## Barriers & Targets

### 2006 MYRDDP Section 3.6.4.1 Targets:

- Provide expertise and technical data on hydrogen behavior and hydrogen technologies
- Hydrogen storage tank standards for portable, stationary and vehicular use
- Materials reference guide for design and installation
- Standards for pipelines, delivery and ancillary equipment

### 2006 MYRDDP Section 3.6.4.2 Barriers:

- J. Lack of National Consensus on Codes & Standards
- K. Lack of Sustained Domestic Industry Support at International Technical Committees
- N. Insufficient Technical Data to Revise Standards



# Objectives

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- Safe design of structures for storage and transport of high-pressure hydrogen gas requires material property data that reflects service conditions
  - Identify and document existing data on hydrogen compatibility of materials from technical journals and reports
  - Generate new data through materials testing, emphasizing testing in high-pressure hydrogen gas
- Provide advocacy and technical support for the codes and standards change process
  - ASME Project Team on Hydrogen Tanks
  - ASME Project Team on Hydrogen Piping and Pipelines
  - CSA HGV and HPRD technical committees



# Approach

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- Compose “Technical Reference for Hydrogen Compatibility of Materials”, a compilation of existing data in material-specific chapters ([www.ca.sandia.gov/matlsTechRef](http://www.ca.sandia.gov/matlsTechRef))
  - ferritic steels (pipelines and pressure vessels)
  - stainless steels (tubing and valves)
  - aluminum alloys (pressure vessels and components)
  - copper (seals)
  - materials not recommended for H<sub>2</sub> service (ferritic stainless steels)
- Conduct materials testing in H<sub>2</sub> gas up to 100 MPa pressure
  - Test relevant structural materials, selected in collaboration with SDOs and industry stakeholders
  - Emphasize fracture mechanics methods
  - Establish procedures for generating reliable design data



# Technical Reference for Hydrogen Compatibility of Materials

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- Version 1 completed
  - 14 chapters posted to website (May 2007)
- 4 chapters completed over this past reporting period
  - carbon steels (pipelines)
  - aluminum (pressure vessels and components)
  - duplex stainless steels (tubing and valves)
  - ferritic stainless steels (not recommended for H<sub>2</sub> service)
- New chapters scheduled for Version 2
  - heat treatable aluminum (pressure vessels and components)
  - 310 and 321 stainless steels (tubing and valves)
  - martensitic stainless steels (not recommended for H<sub>2</sub> service)

New chapters in Version 2 motivated by needs and interest communicated by stakeholders



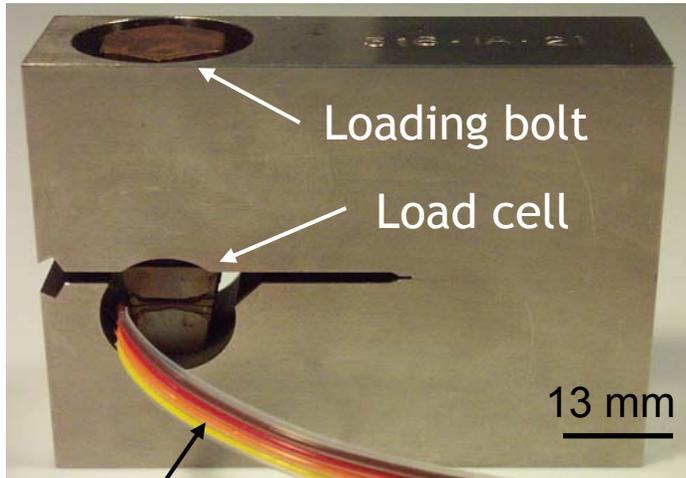
# Testing steels for pressure vessels

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- ASME Boiler and Pressure Vessel Project Team on Hydrogen Tanks created Article KD-10 for Section VIII, Division 3
  - “Special Requirements for Vessels in High Pressure Gaseous Hydrogen Service”
  - Specifies need for fracture mechanics-based materials data (i.e., cracking thresholds and fatigue crack growth rates) in high-pressure hydrogen gas up to 100 MPa
- Two objectives for materials testing:
  - Generate benchmark H<sub>2</sub> cracking thresholds for low-alloy steels currently in codes for seamless pressure vessels (SA 372 Gr. J, DOT 3T, DOT 3AAX)
  - Establish best procedures for testing in H<sub>2</sub>

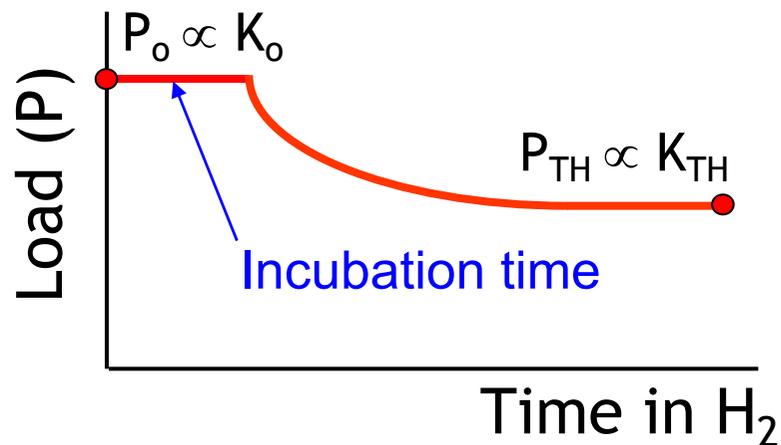
# Measuring benchmark cracking thresholds ( $K_{TH}$ )

wedge opening load (WOL)  
cracking threshold specimen

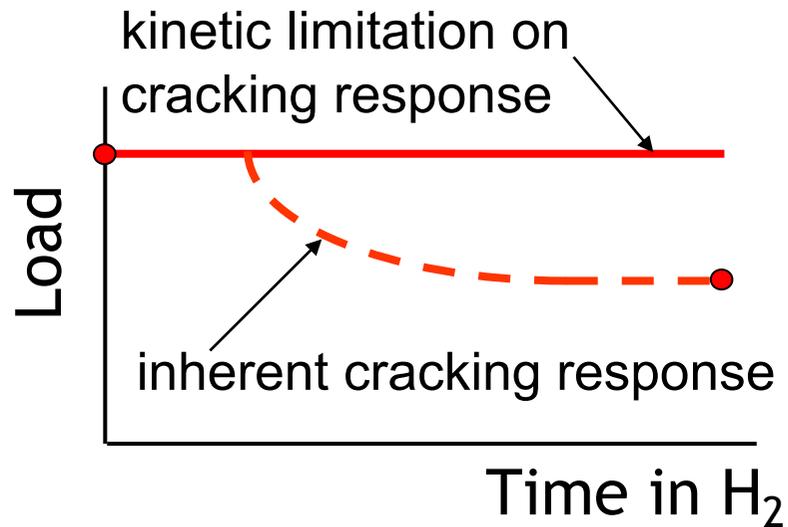


strain gage leads (Excitation and DAQ)

- Load applied with bolt, load measured *in situ* with load cell
- Specimen bolted to  $K_0 > K_{TH}$  in air, then exposed to  $H_2$  gas
- Load cell gives load vs. time: crack advance  $\rightarrow$  load drop
- Crack propagation initiates after **incubation time**, arrests when  $K = K_{TH}$



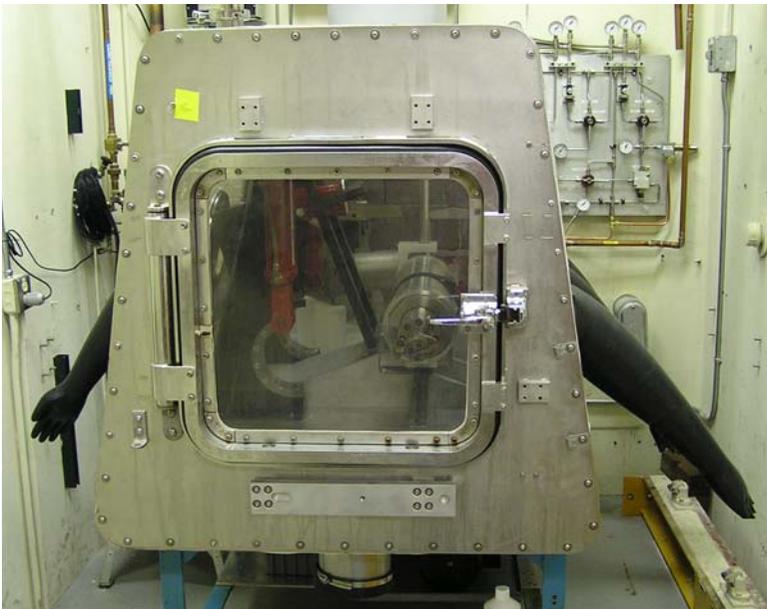
# Long incubation times during cracking threshold tests circumvented with glove box



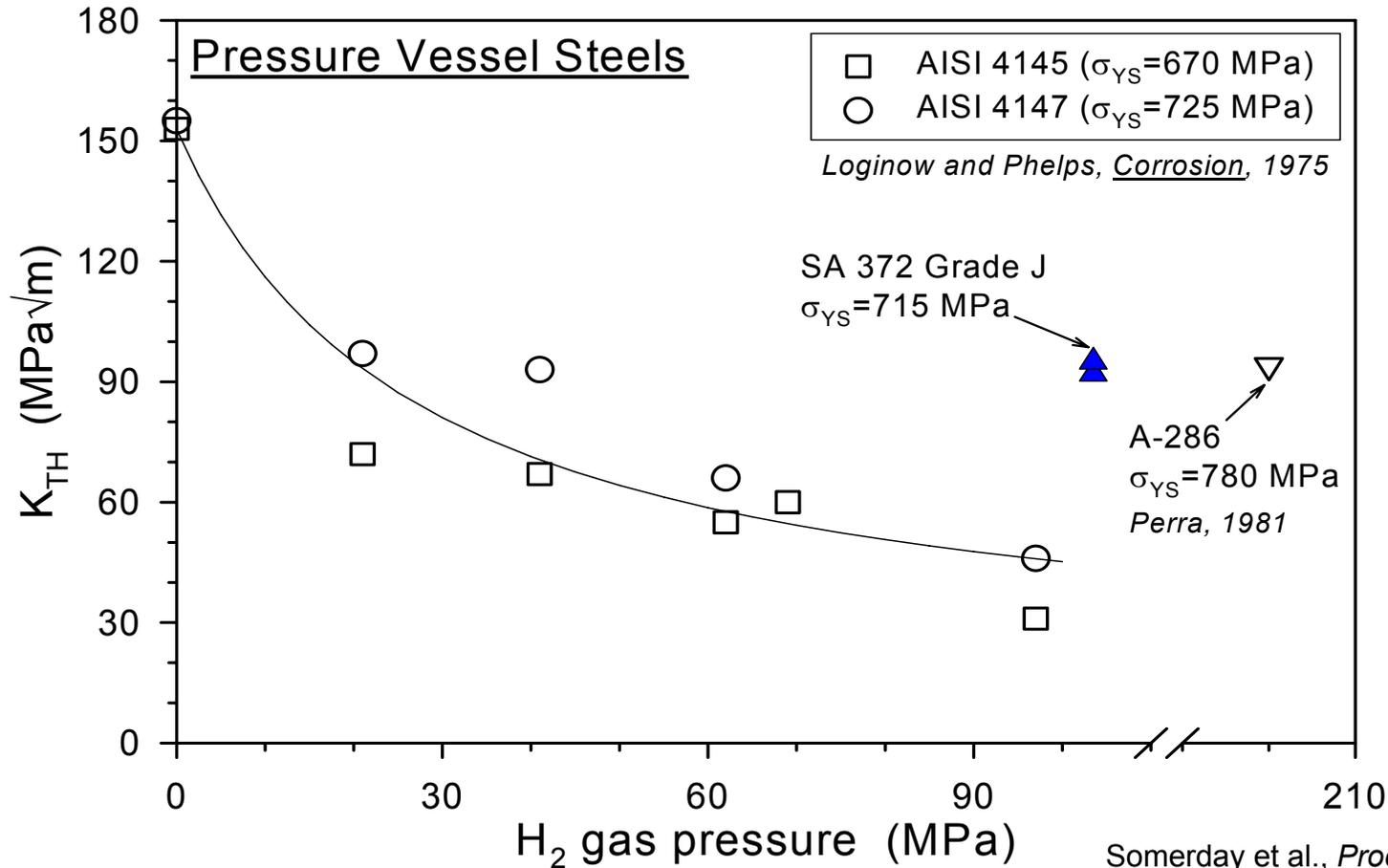
- Initial tests on steel specimens loaded in **air** had extended incubation times
  - Incubation times could be **> 5000 hr**
  - **Can give false indication of “immunity” to cracking in H<sub>2</sub>**

- Designed custom glove box with internal fixtures for loading specimens in **1 ppm O<sub>2</sub> environment**
  - Incubation times **< 24 hr**

Environment control procedures included in ASME Article KD-10



# Cracking threshold ( $K_{TH}$ ) measured for SA 372 Gr. J in 100 MPa $H_2$



Somerday et al., *Proc. International Hydrogen Energy Development Forum & Workshop*, 2007

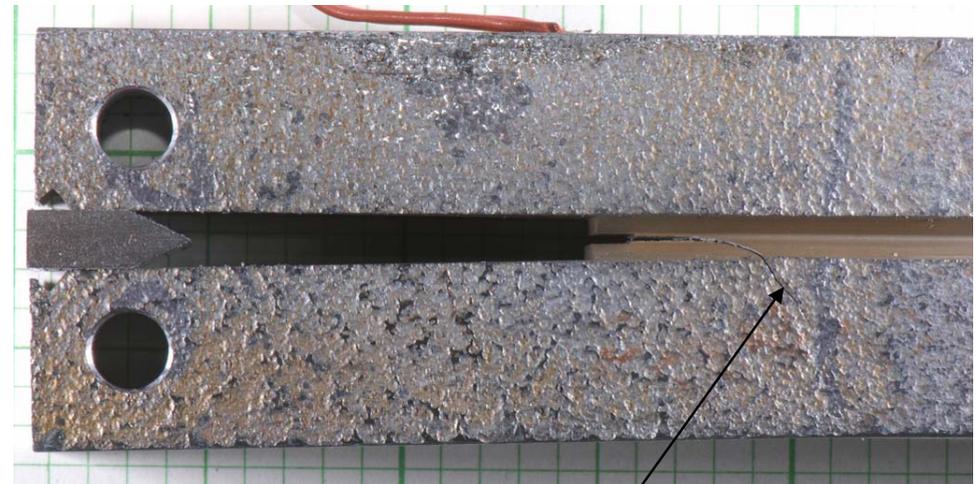
$K_{TH}$  values for SA 372 Gr. J higher than historical values and approach values for stainless steel A-286

# Cracking threshold specimen design must be selected judiciously

steel panel from vessel



double-cantilever beam (DCB)  
specimen of DOT 3T steel



deflected crack

- DCB specimen geometry may not yield successful results for materials with high  $H_2$  cracking thresholds
  - Solution: design curved WOL specimen for testing materials from vessel/pipe walls

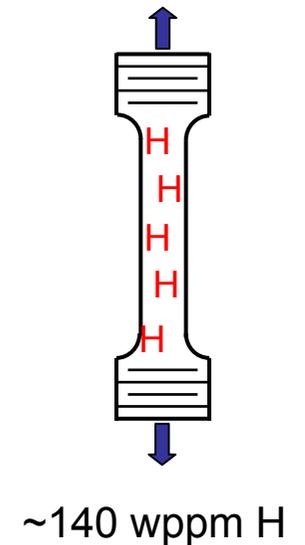
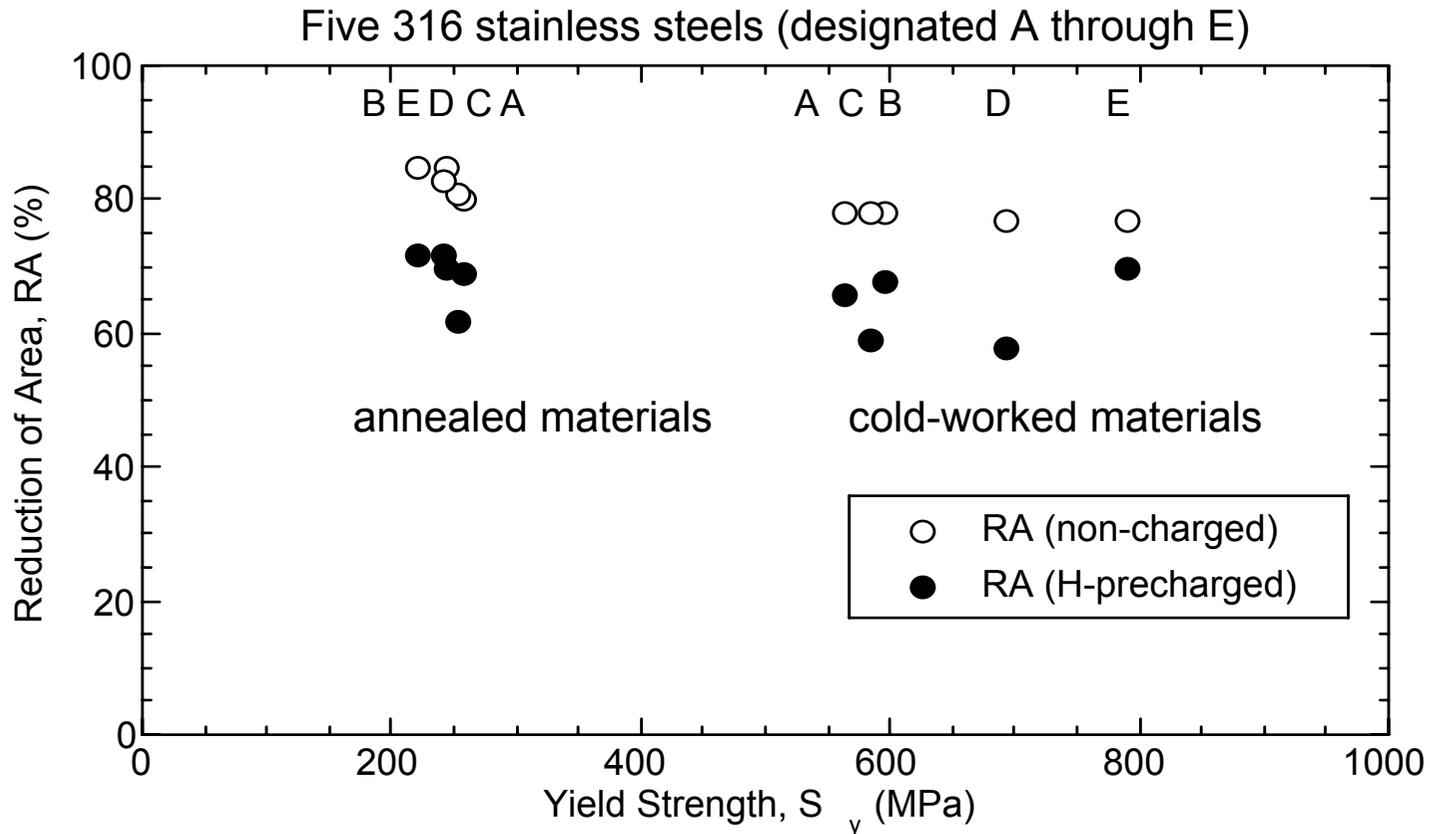


# Testing stainless steels for piping

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- ASME Project Team on H<sub>2</sub> Piping and Pipelines creating new code B31.12
  - Need material property data for 316 stainless steel exposed to high-pressure hydrogen gas
- Sandia role guided by Materials for the Hydrogen Economy Coordinating Group and DOE Pipeline Working Group
- Generate **benchmark tensile fracture data** of 316 stainless steel after hydrogen gas exposure
  - Emphasize cold-worked microstructures, varying test temperature, and effect of stress concentrations

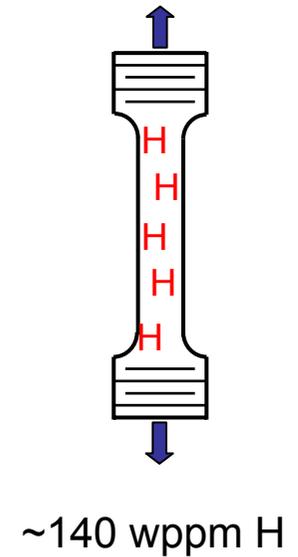
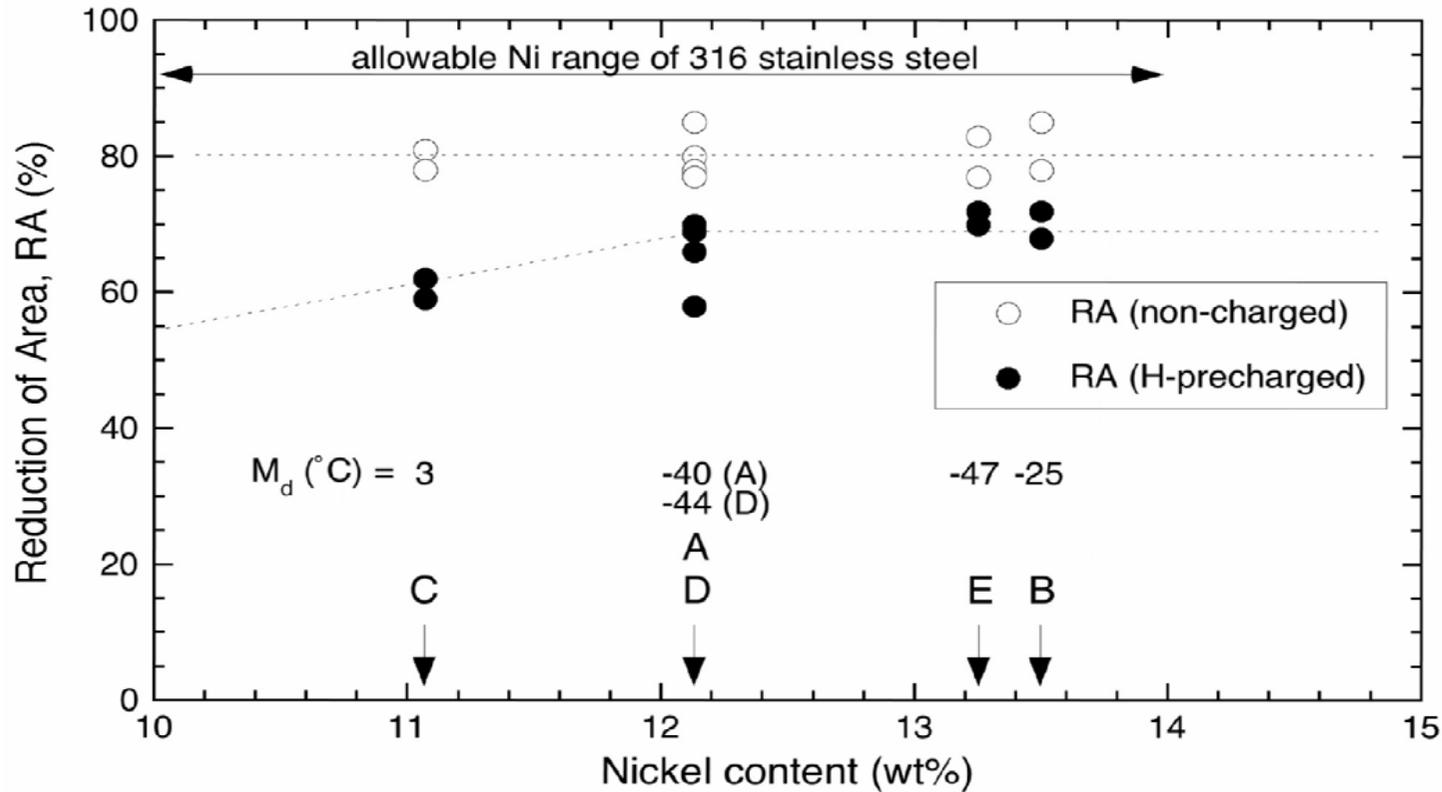
# Tensile fracture of H<sub>2</sub>-exposed 316 stainless steels as a function of cold work



San Marchi et al., submitted to  
*Int J Hydrogen Energy*, 2007

Tensile fracture resistance of cold-worked (high-strength)  
316 similar to annealed material

# Tensile fracture of H<sub>2</sub>-exposed 316 stainless steels as a function of Ni content

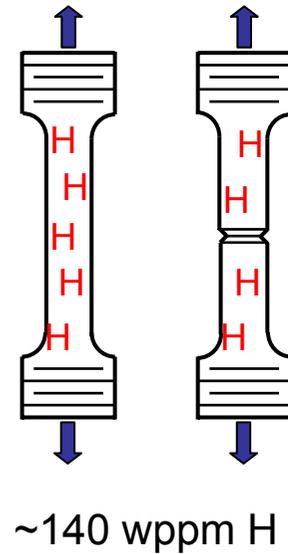
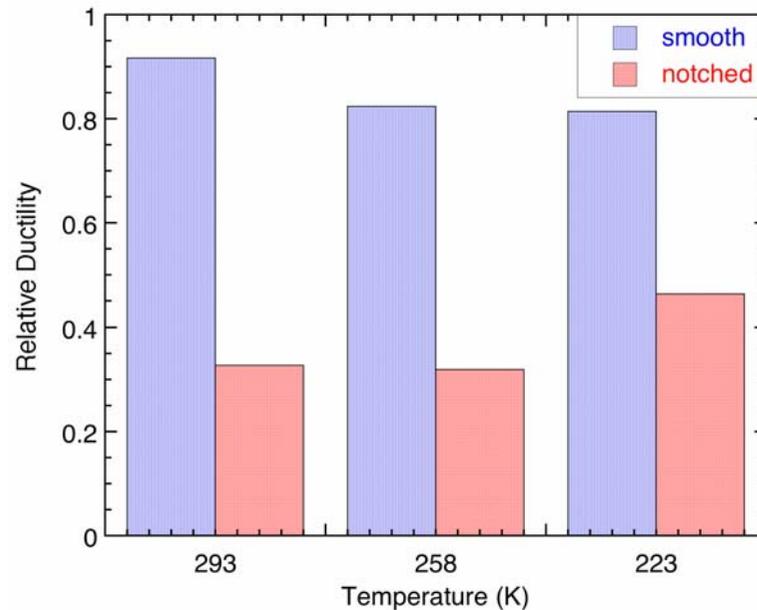
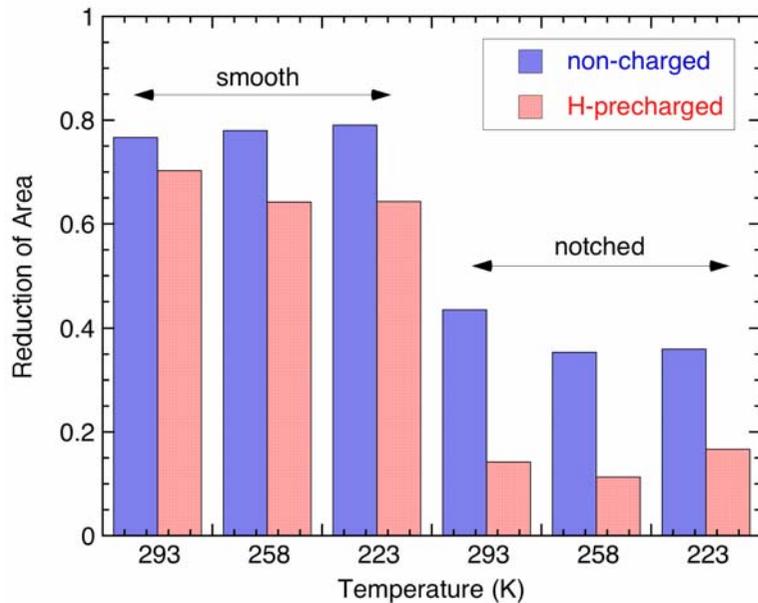


San Marchi et al., submitted to  
*Int J Hydrogen Energy*, 2007

H<sub>2</sub> compatibility of 316 stainless steel can be optimized by controlling composition, particularly nickel

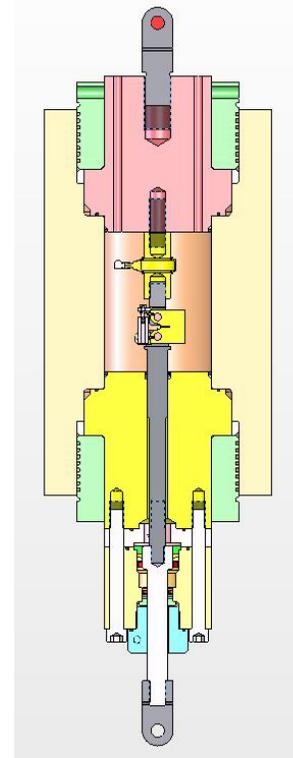
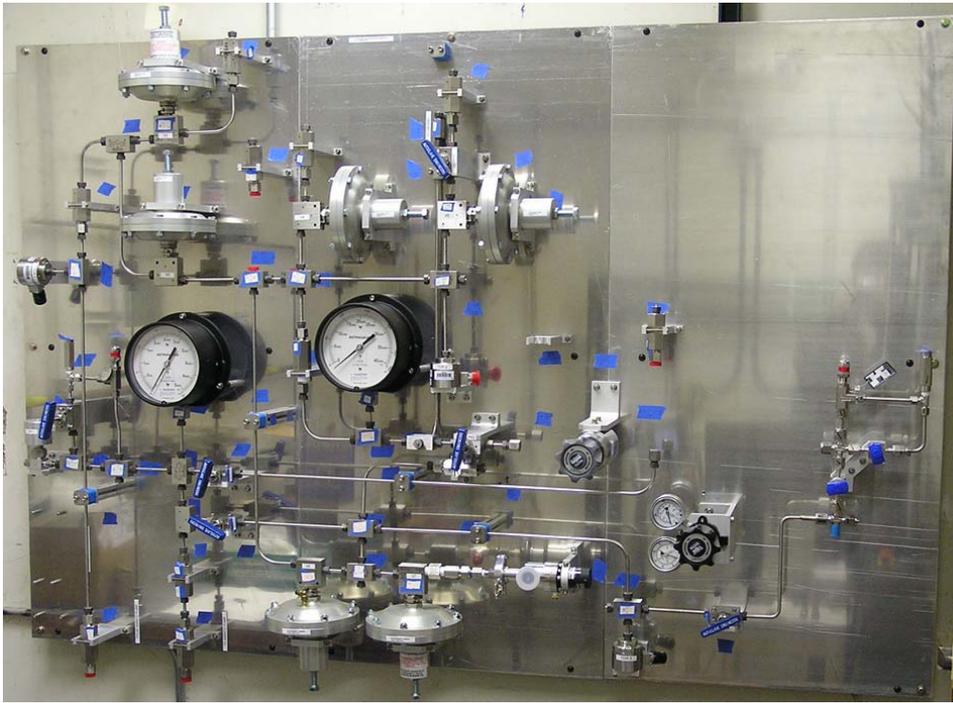
# Tensile fracture of 316 as a function of temperature and stress concentration

Cold-worked 316 stainless steel Alloy E



$H_2$  compatibility of 316 stainless steel depends mildly on temperature but strongly on stress concentration

# Design of system for fatigue crack growth tests in high-pressure H<sub>2</sub> gas



- ASME Article KD-10 requires fatigue crack growth rate data for hydrogen containment structure design
- Pressure vessel delivery delayed by feed-through subcontractor
- Only system in U.S. for conducting fatigue tests in 100 MPa hydrogen gas



# Future Work

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## Remainder of FY07

- Measure cracking thresholds of DOT 3T and DOT 3AAX steels in 100 MPa H<sub>2</sub>
- Complete data set for SA 372 Gr. J steel in 100 MPa H<sub>2</sub> to satisfy Article KD-10 (i.e., measure fatigue crack growth and cracking thresholds at low temperature)
- Initiate next phase of 316 stainless steel testing in collaboration with industrial partner
- Start work on version 2 of Materials Technical Reference

## FY08

- Create data set for Cr-Mo-Ni steel (e.g., SA 372 Gr. M) in 100 MPa H<sub>2</sub> to satisfy Article KD-10 (i.e., fatigue crack growth and cracking thresholds)
- Continue testing of 316 stainless steels (e.g., testing in fatigue, testing of welds)
- Compose new chapters for version 2 of Technical Reference (e.g., stainless steels 310, 321, 17-4 PH)



# Summary

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- Completed Technical Reference Version 1 containing existing data on high-priority structural materials
- Measured fracture properties of hydrogen-exposed steels used in pressure vessels and piping
  - cracking threshold of SA 372 Gr. J in 100 MPa H<sub>2</sub> gas about three times higher than historical data
  - tensile fracture of 316 not affected by cold work, but can depend on nickel content and stress concentration
- Finalizing laboratory infrastructure for fatigue crack growth testing in high-pressure H<sub>2</sub> gas